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RAPID RADIOCHEMICAL PROCEDURE FOR
ANTIMONY AND ARSENIC

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ADMINISTRATIVE INFORMATION

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ABSTRACT

A very rapid radiochemical procedure has been developed for antimony and arsenic. A sample is added to a 30 per cent solution of H_2SO_4 containing a carrier for antimony or arsenic (or both). This solution is dropped into a flask containing granular zinc at $100^\circ C$. The volatile stibine and/or arsine formed is passed through a hot quartz tube and decomposed to the metal, which collects as a metallic mirror on a cooler portion of the quartz tube. This metal is dissolved in H_2SO_4 and assayed. The chemical yield is consistently between 70 and 80 per cent. The time required for separation of the metal from the other elements of a fission product solution is about 10 seconds. The decontamination factors for the antimony procedure are: Mixed Fission Products (2 weeks old) = 10^5 , I = 3×10^4 , Te = 4×10^4 , Sn = $> 10^5$, As = 20-50. The decontamination factors for the arsenic procedure are: Mixed Fission Products = 10^7 , Sb = 2×10^3 , Sn = $> 10^6$.

SUMMARY

The Problem

Rapid radiochemical procedures for antimony and arsenic fission products do not exist. The problem was to develop a radiochemical procedure for antimony and arsenic that would take less than 1 minute to separate them from their parent and daughter nuclides.

Findings

A high-chemical-yield, 10-second radiochemical separation of antimony and arsenic fission products from their parent and daughter nuclides with decontamination factors $> 10^4$ has been developed. It involves a rapid distillation of their hydrides, followed by a thermal conversion to the metallic state.

INTRODUCTION

A rapid, one-step radiochemical method of separating antimony from fission products was needed for the determination of independent fission yields of antimony nuclides. The separation must be completed before the antimony decays, and the exact time of separation after fission must be known. The brief half-lives (minutes or less) preclude oxidation-reduction reactions to effect isotopic exchange and repeated precipitation to achieve the required radiochemical purity. Also, the decontamination factors from tin, tellurium, and iodine must be large, since the independent fission yield of the antimony nuclide is finally determined from the number of atoms of the longer-lived iodine daughters.

Present radiochemical procedures for separation of antimony and of arsenic^{1,4} include solvent extraction, ion exchange, reduction to the metal, sulfide precipitation, hydride formation, and combinations of these methods. The time for separation varies from five minutes to five hours. Chemical yields vary from 25 to 90 per cent. Decontamination factors for individual elements vary widely and usually are from 10^2 to 10^4 .

Historically, small quantities of arsenic and antimony have been determined by the Marsh test,³ or a simple variation of it, in which arsine (or stibine) is formed by the reduction with zinc in hydrochloric acid. This method promised to meet the requirements, provided the relatively low yield always obtained could be increased and the antimony and arsenic could be collected without contamination by iodine and other volatile fission products. The method was modified in the separation of the volatile hydrides of antimony and arsenic, and was developed into the desired radiochemical procedure. By using heated reactants the volatile hydride formed quickly and was swept from the solution by evolved hydrogen before it decomposed. This was the main method of increasing the yield from about 15 per cent to 80 per cent. Also, a filter system was added to remove the gaseous fission products and the contaminated aerosol carried by the evolved hydrogen gas.

While this procedure was developed for the separation of a specific nuclide from a fission product mixture, its use is by no means so limited. Carrier-free separations are achieved by simply omitting the carriers. The procedure has been used for quickly separating antimony activities at a well-defined time from mixtures of antimony and its radioactive daughters. Also, antimony can be rapidly determined through isotopic dilution, with radioactive antimony used for the radioisotopic yield determination.

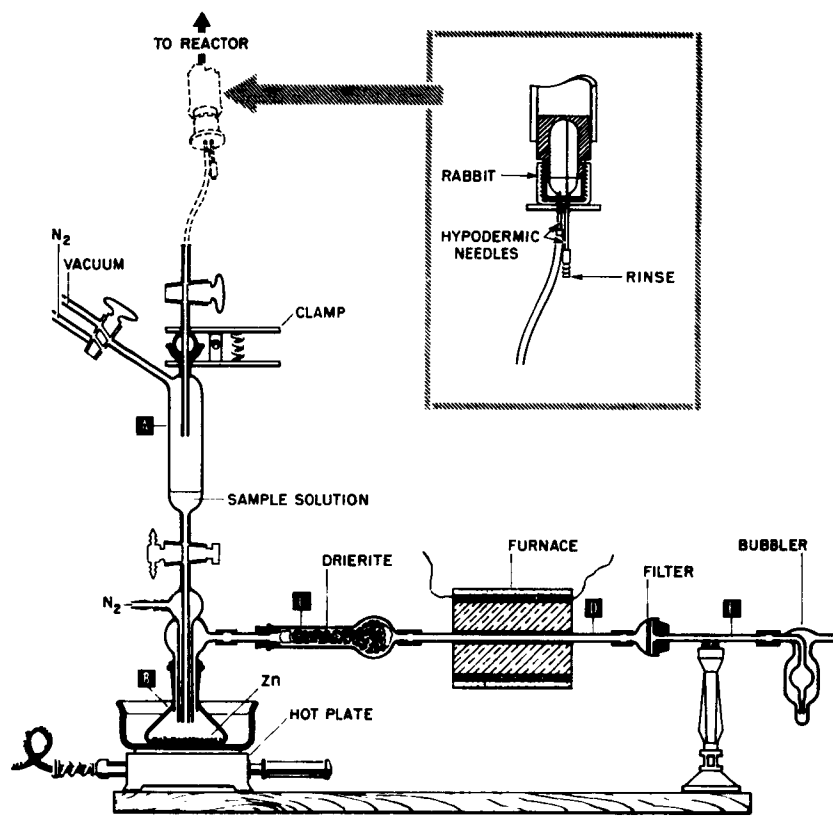


Fig. 1 Apparatus for Separation of Antimony and Arsenic

EXPERIMENTAL

Materials

Sulfuric acid, specific gravity 1.84, reagent, low arsenic.
30 per cent sulfuric acid, reagent, low arsenic.
Antimony carrier, 10 mg Sb/ml as SbCl_3 , reagent.
Arsenic carrier, 10 mg As/ml as NaAsO_2 , reagent.
Zinc metal, granular, 20 mesh, reagent, low arsenic.
Drierite desiccant, 8 mesh (anhydrous CaSO_4).
Glass wool.

Apparatus

The apparatus for the determination of antimony and arsenic is shown in Fig. 1. Unit A holds the solution of acid, carriers, and mixed fission products (MFP). It is fitted with a two-way stopcock to provide vacuum for introducing the MFP into the vessel, and nitrogen for flushing air out of the unit and forcing the solution rapidly into flask B. Unit B is a flat-bottom 70 ml pyrex flask with a 19/38 $\frac{1}{8}$ joint that fits onto Unit A. A 750-watt thermostatically controlled hot plate is used for heating the zinc to 100°C (via the water bath, and flask B). Drying tube C, 12 mm I.D. X 100 mm long, contains Drierite. A 1320-watt tube furnace, connected to a variable transformer, is used to heat quartz tube D, 4 mm I.D. X 20 mm long, to 600°C. A fine sintered glass filter is located between quartz tube D and E to catch the fine particulate black antimony which forms from the rapid cooling of antimony vapor. A Meker -type burner is used to heat quartz tube E for the decomposition and collection of arsine. A bubbler placed after quartz tube E indicates the flow rate of the flushing gas. Finally, tubing is attached to the bubbler to lead the escaping gases to a well-ventilated hood. Extreme care should be taken to prevent escape of the very poisonous arsine and stibine from the system.

Procedure for Antimony and Arsenic

The apparatus is shown in Fig. 1. The furnace temperature is held at 600°C by adjustment of a variable transformer. The second quartz tube E is heated until it has a reddish glow. Five grams of zinc metal is spread evenly over the flat bottom of flask B, and held at a temperature of 100°C in the water bath. Flask B and the system are flushed for a few minutes with an inert gas (e.g., N_2) before starting the analysis. The gas inlet for flushing is shown just under the bottom stopcock of A, Fig. 1. Four milliliters of 30 per cent

H_2SO_4 , one milligram each of antimony and arsenic carriers, and the MFPs are added to Unit A. The unit is flushed for a few seconds with an inert gas by way of the three-way stopcock and then put under a few pounds pressure. The solution in A is then added to the zinc in B. Within about 10 seconds, the antimony metal deposits on quartz tube D and the sintered glass filter, and the arsenic on the hotter quartz tube E. The collected sample is dissolved from the tubes and the filter with warm concentrated H_2SO_4 . Other reagents may be used for this solution step. The chemical yields may be determined by any convenient method.

Procedure for Antimony

To analyze only for antimony, the apparatus used is that shown in Fig. 1 except for the furnace and quartz tube E. Five grams of zinc metal is spread evenly over the flat bottom of flask B, and held at 100°C in the water bath. Flask B and the system are flushed for a few minutes with an inert gas before starting the analysis. Four milliliters of 30 per cent H_2SO_4 , one milligram of antimony carrier are added to Unit A. The unit is flushed for a few minutes with an inert gas by way of the three-way stopcock. Quartz tube D is heated with the Meker-type burner until it has a reddish glow. The sample is introduced as before in the Antimony-Arsenic procedure or introduced with a transfer pipet. After mixing, the air is flushed out by the inert gas. Flask A is put under a few pounds pressure to force the reagents into B. The antimony metal deposits on quartz tube D and the sintered glass filter. The metal dissolves easily in warm concentrated sulfuric acid. If it is desirable to study the decay products of antimony, the metal can be dissolved in tartaric acid with tellurium or iodine carrier present. The chemical yield can be determined by the use of a spectrophotometric method using the Rhodamine B colored complex formed with antimony⁵ or with an isotope tracer (e.g., Sb^{124}).

In this separation the arsenic deposits with the antimony. Since the fission yield of arsenic is low compared to antimony and the half-lives of arsenic fission product nuclides are short compared to those of antimony fission product nuclides, this contamination by arsenic is insignificant in the usual antimony analysis performed on fission product samples several days old. By using the procedure for both antimony and arsenic, the arsenic contamination is less than five per cent.

RESULTS AND DISCUSSION

Application

In making rapid separations of antimony radionuclides from mixed fission products, a pneumatic system carried the fission products rapidly from the reactor to the apparatus where the separation of antimony was to be made. A "rabbit" holding the fission product solution and antimony carrier in 30 per cent H_2SO_4 impales itself onto a hyperdermic needle and the solution is drawn into A, along with a 1-ml 1N HCl wash, (Fig. 1) by means of a vacuum.² After a brief mixing, the fission products, 30 per cent H_2SO_4 , and antimony carriers (inactive antimony plus Sb^{124}) are dropped onto zinc in B, where SbH_3 is formed. The stibine is swept out by the generated H_2 and passes through the Drierite in C, which picks up any spray from the reaction in B. This trap also is very efficient in preventing the volatile fission products, such as I_2 which comes over as SbI_3 , from passing through the system. The SbH_3 is decomposed by passing through the hot quartz tube D and deposits as an antimony mirror on the cold portion of the quartz tube and as black antimony on the sintered glass filter. In the determination of the Sb^{133} independent fission yield, the antimony was given time (\sim one day) to decay completely to 21 hour I^{133} . The antimony metal was dissolved in tartaric acid containing iodine carrier instead of hot H_2SO_4 so that none of the iodine would be lost by volatilization. A radiochemical determination was then carried out for iodine. All the I^{133} present had decayed from the Sb^{133} . The chemical yield of antimony was determined by counting the Sb^{124} , which was added for this purpose. Fig. 2 shows the results of a series of separations of antimony at various times after a 10-second irradiation of U^{235} using the method described. The half-life of Sb^{133} was found to be 2.6 min. The independent fission yields of Sb^{133} , Sb^{132} and Sb^{131} from thermal neutron irradiation of U^{235} were found to be 2.92, 2.04, and 2.81 per cent, respectively.

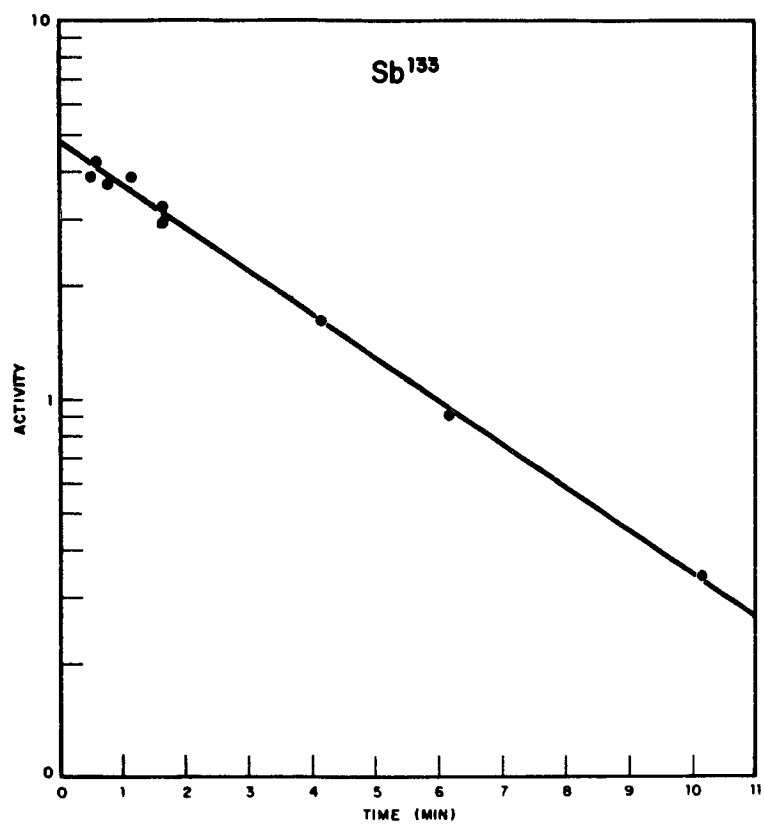


Fig. 2 Amount of Sb^{133} Separated at Various Times After Fission

Other Uses

In the course of development of this procedure it was apparent that the use of carriers was not essential, and it could thus be used for carrier-free preparations of these elements. Since the deposition characteristics of the antimony and arsenic can be determined with carriers, the carrier-free material is leached from the same position on the tube where the carriers would normally deposit.

This procedure is also useful when it is desirable to separate antimony activity from a mixture of its radioactive daughters. The collection period takes only a few seconds and the time of separation can be determined very accurately. This is of value when making a correction for or a study of the growth and decay of daughter activities.

Using this procedure for the determination of antimony in mineral or other samples is possible by isotopic dilution techniques. One may dissolve the sample in sulfuric acid, collect the metallic antimony on the quartz tube, dissolve it, and determine the quantity of antimony spectrophotometrically. The yield is determined by the added radioactive antimony whose contribution to total antimony is insignificant. This method is faster than present analytical methods for antimony. These techniques may also be applied to the determination of arsenic.

Study of Variables

Since the major weakness of procedures using separations of stibine and arsine is low yield, the major effort in developing this procedure was concentrated on this problem. It was found that the yield could be improved by reducing the normal carrier concentration. One milligram of carrier gives a satisfactory yield and is enough to give good results with the spectrophotometric method used in the measurement of chemical yield.

The chemical yield is also dependent on the total volume of reagents (in the apparatus used). A total volume of five milliliters seemed optimum with four milliliters of this being 30 per cent H_2SO_4 . Since a 10 mg/ml standardized carrier solution was used, there remained 0.8 to 0.9 milliliter for sample solution. If 1.2 ml of concentrated H_2SO_4 is used instead of four milliliters of the 30 per cent acid,

3.6 to 3.7 ml of sample solution can be taken. Variations in total volume of 10 per cent in either direction have little effect on the chemical yield.

The chemical yield is also somewhat dependent on the mass of zinc and its particle size. There is an increase in yield up to a mass of five grams of zinc, but from five to 15 grams there is no noticeable improvement. Thus, five grams was chosen as the best weight of zinc in relation to the other conditions chosen. There is a small increase in yield using 20 mesh instead of 10 mesh particle size zinc. Since still finer particle sizes tend to cause excessive foaming, the 20 mesh size was chosen.

The factor of greatest importance in attaining the maximum yield was found to be the temperature of reagents. Initially the temperature of the solution was raised in order to have the reaction start quickly so that the time for separation could be reduced to a minimum. It was found that increasing the temperature of the solution gave an increased yield as well as a faster separation. A yield of about 25 per cent was the best that could be obtained with this arrangement. Heating the solution led to difficulties in drawing in the fission products rapidly from the rabbit by the use of a vacuum, since the vacuum could not be maintained over a boiling solution. To avoid this difficulty, the zinc and its container were heated. With this simple change in conditions the yield went up to a consistent 70-80 per cent. A temperature of about 100°C was found to be optimum. This temperature is easily maintained through the use of a water bath. The yield was reduced by a third by lowering the temperature to 80°C.

Exchange of carrier with tracer is usually a problem in radio-chemical procedures where the element in question can exist in two or more oxidation states. This problem was studied in the following manner. Sb^{124} (III) was mixed with Sb^{125} (V). The gamma spectrum of the mixture was observed on a 256-channel gamma-ray analyzer. The mixture was then run through the antimony separation procedure, the antimony was collected, and the gamma-ray spectrum of this mixture was compared with the initial mixture. There was no detectable difference in the two spectra, indicating that the oxidation state appears to have no measurable effect upon the yield of the antimony. Thus, it is not necessary to perform a series of oxidation-reduction steps to insure exchange. It must be noted, however, that there is a possibility of $\text{Sb}(0)$ being formed in irradiations under reducing conditions. Since reducing conditions did not exist in these studies, no experimental verification was made of the existence of tracer amounts of $\text{Sb}(0)$. It is very unlikely that $\text{Sb}(0)$ would be formed or exist in the H_2SO_4 solution used here.

Since a clean separation of antimony from iodine was essential for our needs, various traps, such as heated copper and other metals were tried. It was found that the desiccant "Drierite", anhydrous calcium sulfate, did by far the best job in removing the iodine activity which was actually volatilized as antimony iodide. It holds back other volatile fission product activities as well. A glass wool plug just before the desiccant stops most of the spray and enables one to make several runs before changing the desiccant.

Decontamination Factors

In the determination of decontamination factors, a measured quantity of the nuclide to be studied was added to the acid and carriers, and the separation of arsenic or antimony was carried out. The ratio of activity added to that collected on the arsenic or antimony recovered is the decontamination factor. Table 1 lists a number of these which

Table 1

DECONTAMINATION FACTORS

| Contaminant | Arsenic | Antimony |
|---|-----------------|-----------------|
| Te | | 4×10^4 |
| I | | 3×10^5 |
| Sn | $> 10^6$ | $> 10^5$ |
| Mixed Fission Products (2 weeks old) | 10^7 | 10^5 |
| As | -- | 20-50 |
| Sb | 2×10^3 | -- |

were of interest in this work. Generally, decontamination factors for arsenic are better than those for antimony, as expected, since arsenic is collected after antimony in the train.

Reasons for High Yield

The speed and high yield attained with this method compared to older separations using arsine and stibine is felt to be primarily due to the heating of the zinc and the consequent rapid removal of the hydrides from the solution before they decompose, and to the reduction in volume of acid reacting with the zinc. With a large volume of acid, the stibine may be oxidized to the metal before it escapes from the

solution. With a volume of solution just large enough to wet the zinc, the stibine escapes easily, along with the voluminous amount of H_2 produced by the reaction of the hot zinc with the sulfuric acid.

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| <p>Naval Radiological Defense Laboratory USNRDL-TR-607</p> <p>RAPID RADIOCHEMICAL PROCEDURE FOR ANTIMONY AND ARSENIC by A.E. Greendale and D.L. Love 17 Jan 1962 19 p. table illus. 4 refs. UNCLASSIFIED</p> <p>A very rapid radiochemical procedure has been developed for antimony and arsenic. A sample is added to a 30 percent solution of H_2SO_4 containing a carrier for antimony or arsenic (or both). This solution is dropped into a flask containing granular zinc at 100°C. The volatile stibine and/or arsine formed is</p> <p>(over)</p> <p>1. Antimony isotopes (Radioactive)- Separation. 2. Arsenic isotopes (Radioactive)- Separation. I. Greendale, A. E. II. Love, D. L. III. Title.</p> <p>UNCLASSIFIED</p> | <p>1. Antimony isotopes (Radioactive)- Separation. 2. Arsenic isotopes (Radioactive)- Separation. I. Greendale, A. E. II. Love, D. L. III. Title.</p> <p>UNCLASSIFIED</p> |
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